

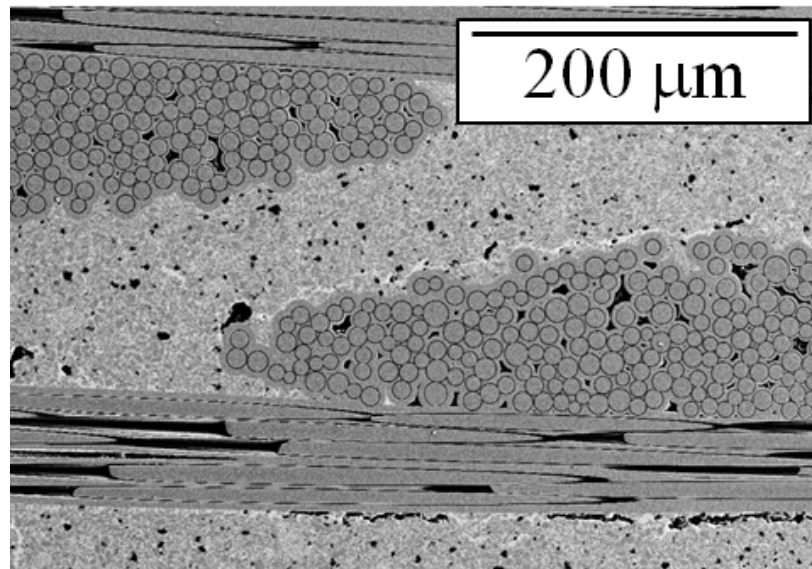
In-Situ SEM Investigation of Microstructural Damage Evolution and Strain Relaxation in a Melt-Infiltrated SiC/SiC Composite

Kathy Sevener, Zhe Chen, Sam Daly - The University of Michigan
Jared Tracy - Stanford University
Doug Kiser - NASA Glenn Research Center

40th Annual Conference on Composites, Materials, and Structures

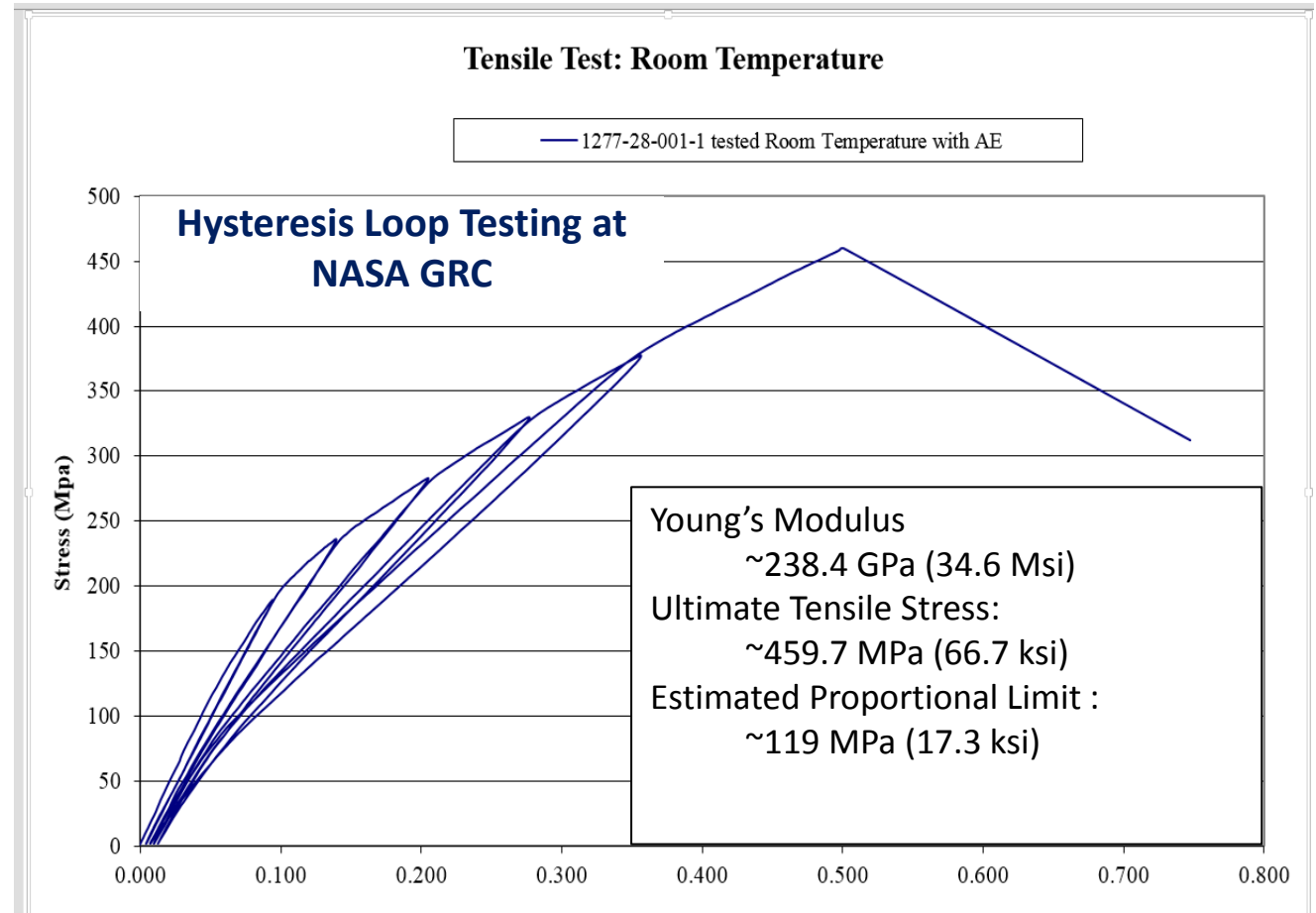
Motivation – Experimental Data for Modeling

- Robust CMC life prediction capabilities require experimental data for inputs and validation
- Several groups working on environmental degradation models that incorporate matrix cracking and interface debonding
 - few studies report measured crack opening displacements
- NASA GRC characterizing CMCs to support environmental modeling



- Sylramic fiber reinforced, slurry cast MI SiC/SiC
- CODs predicted to be very small – too small for traditional DIC
- Apply SEM-DIC using small tensile loading stage in SEM

Approach



- Load in 5 ksi increments to 30 ksi using small tensile stage
- Measure COD using SEM-DIC and manual methods

Digital Image Correlation

Non-contact “optical” method

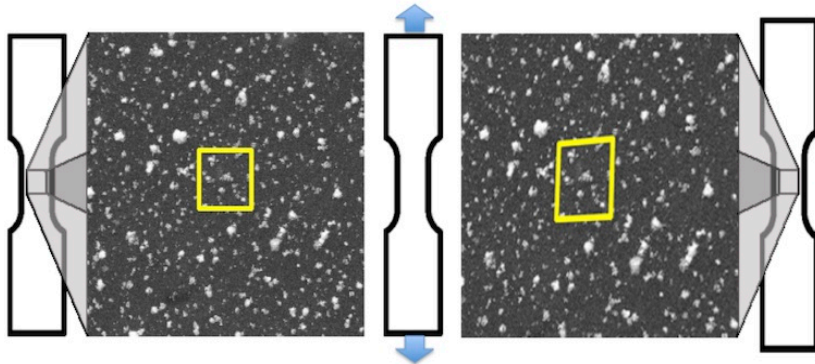
White light, SEM, AFM

Requires surface to have a random tracking pattern

Isotropic, high-contrast, random

Surface pattern analyzed in small subsets

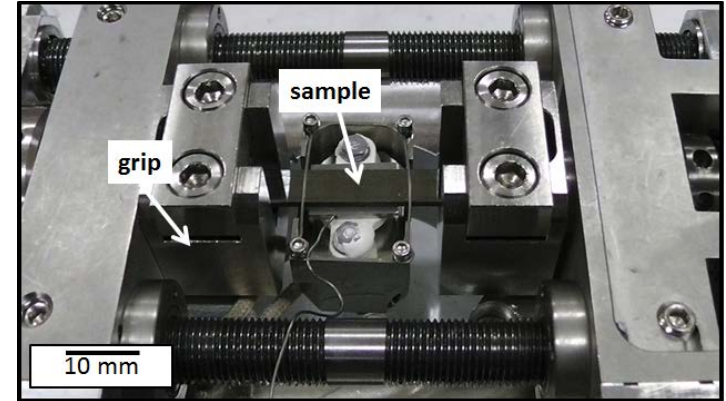
Grayscale intensity within subsets is tracked as sample is deformed.



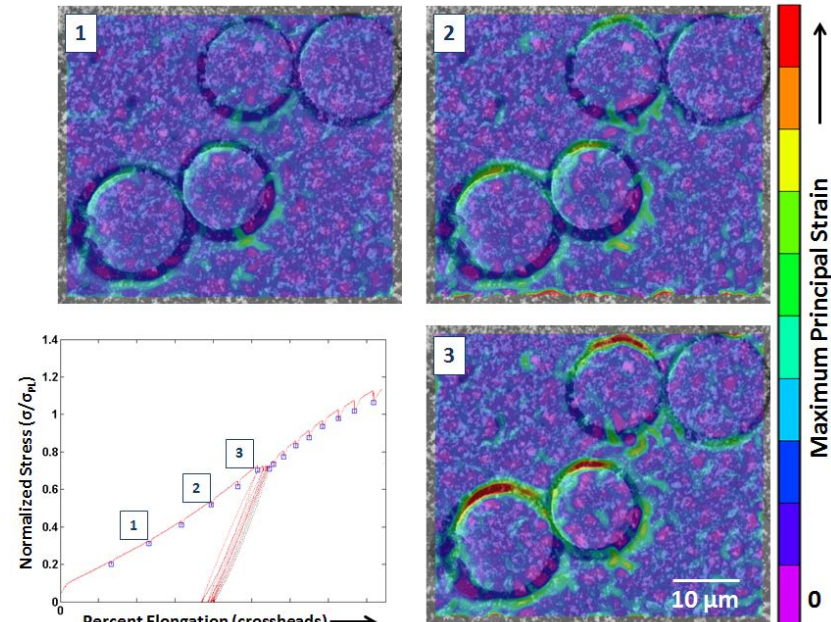
Sutton M.A., et al. (1983), *Image and Vision Computing*, **1**(3): 133-139.
Bruck, H.A., et al. (1989), *Exp. Mech.*, **29**: 261-267.



In-SEM Miniature Tension/Compression Stage

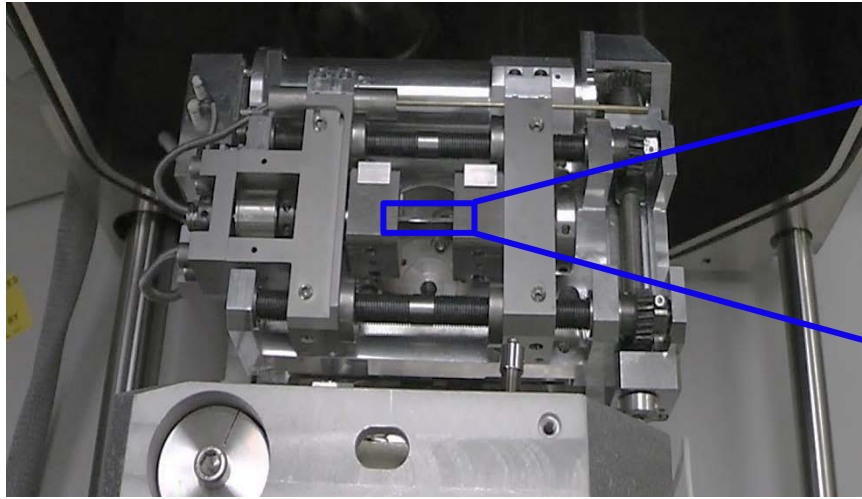


SEM-DIC applied to CMCs



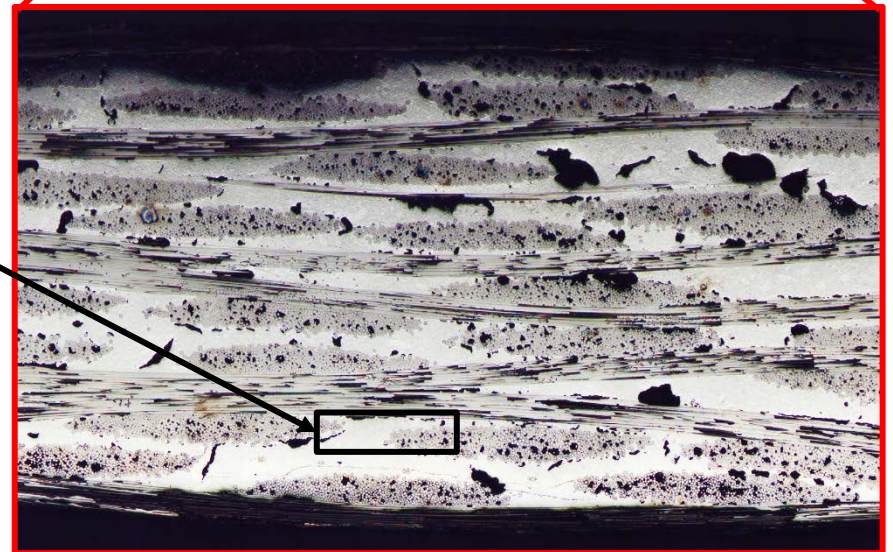
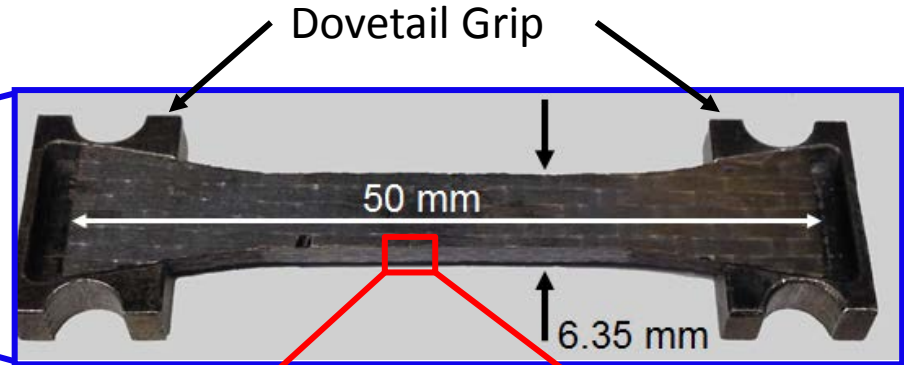
Tracy, J.M., et al. Cocoa Beach 2014

Loading and Imaging Configuration

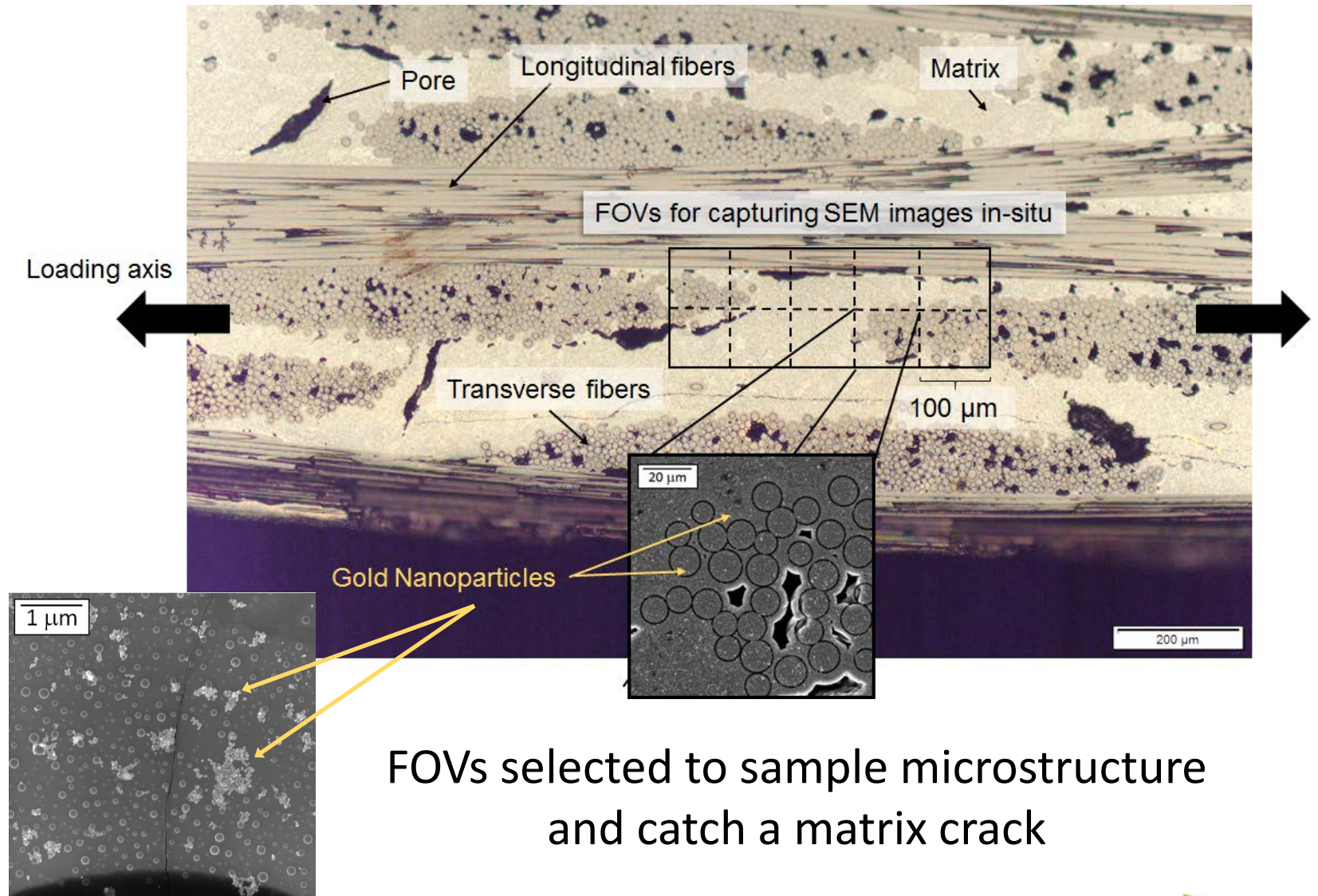


← Loading Direction →

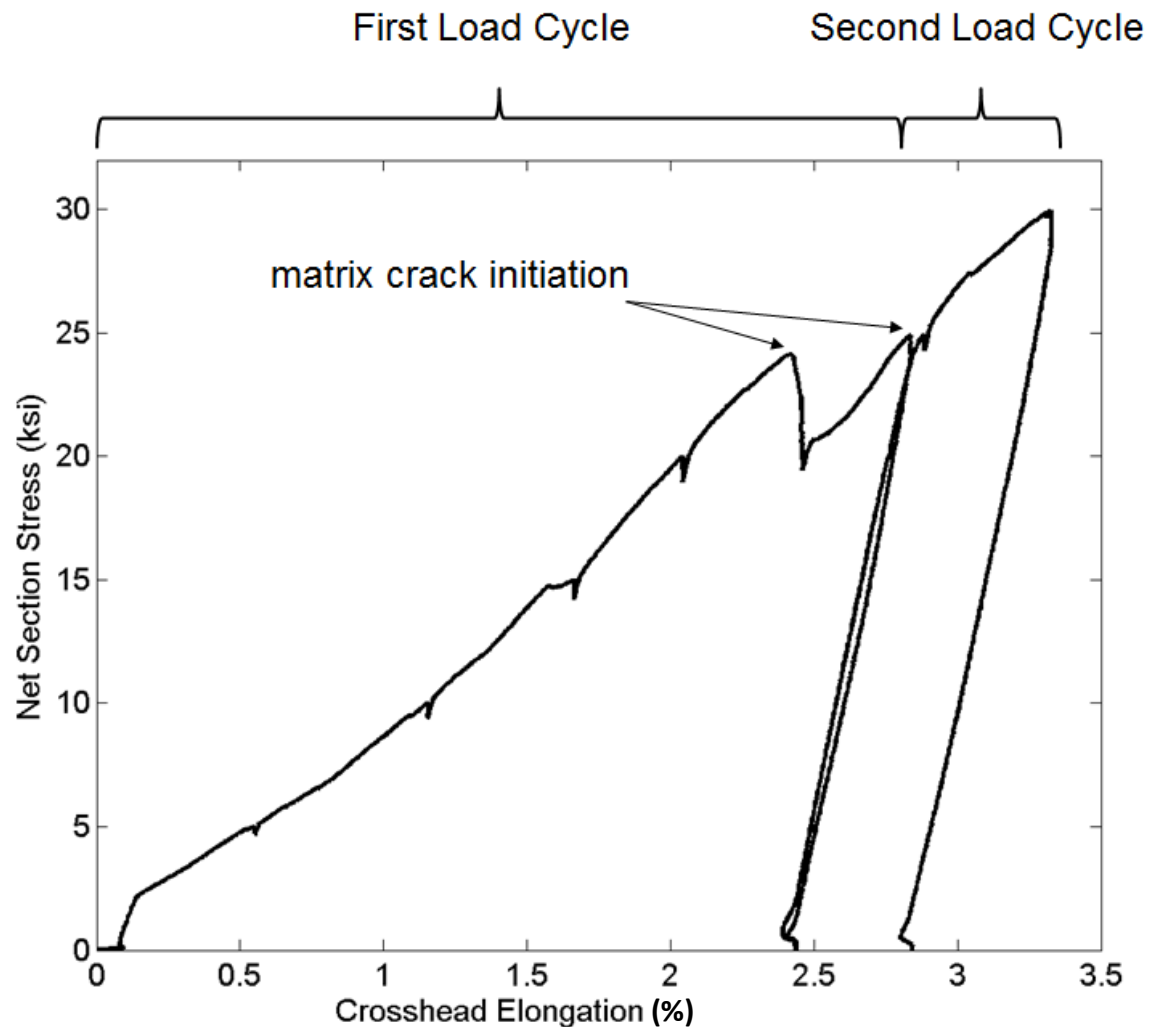
Area analyzed was a $200\ \mu\text{m} \times 500\ \mu\text{m}$ rectangle located $\approx 1.4\ \text{mm}$ left and $0.7\ \text{mm}$ below centroid of gage section



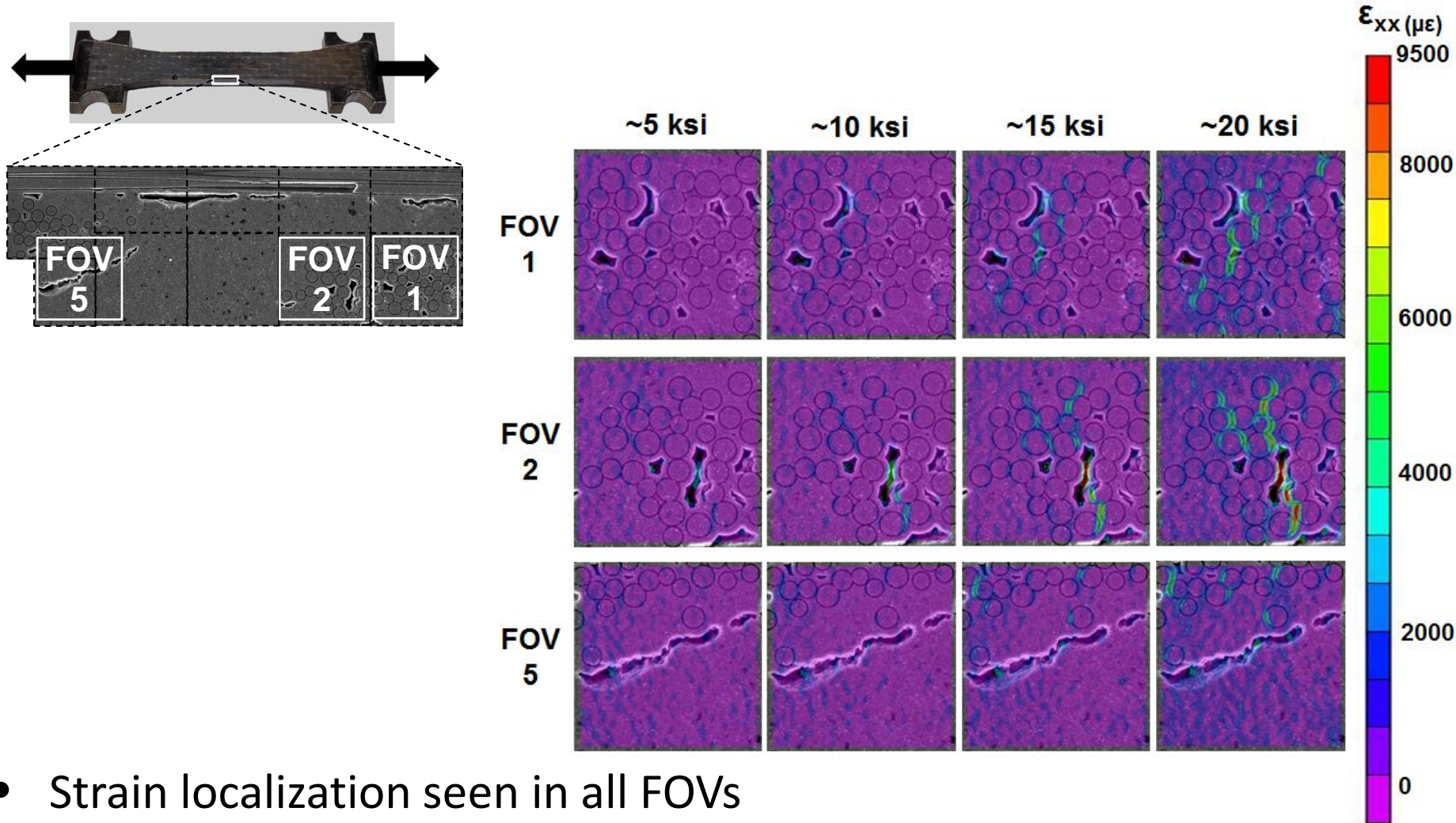
Initial Fields of View (FOVs)



- Sample loaded in tension at ~5 ksi stress increments
- Loading paused at each stress increment to capture SEM images
- Images captured after load relaxed
- Matrix cracks formed between 20 and 25 ksi of initial load cycle, but outside of imaging area.
- Sample unloaded/reloaded to capture matrix crack openings displacements in new AOI



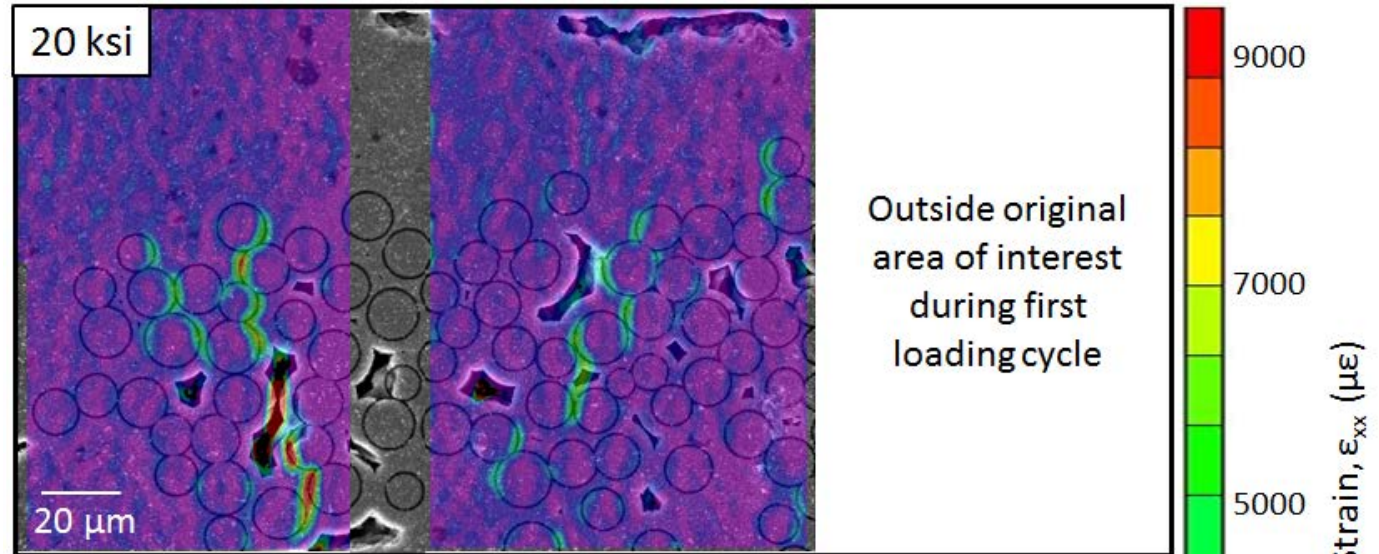
Damage Evolution Before Matrix Cracking



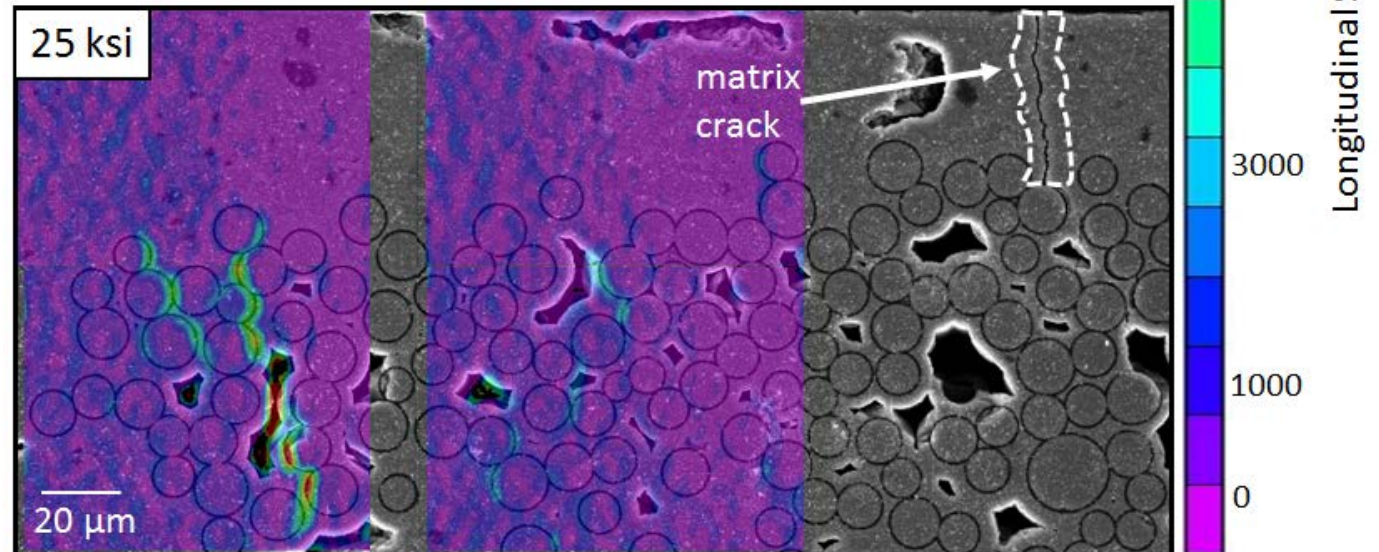
- Strain localization seen in all FOVs
- Strain localization observed ~ 10 ksi

Strain Relaxation Adjacent to Matrix Crack

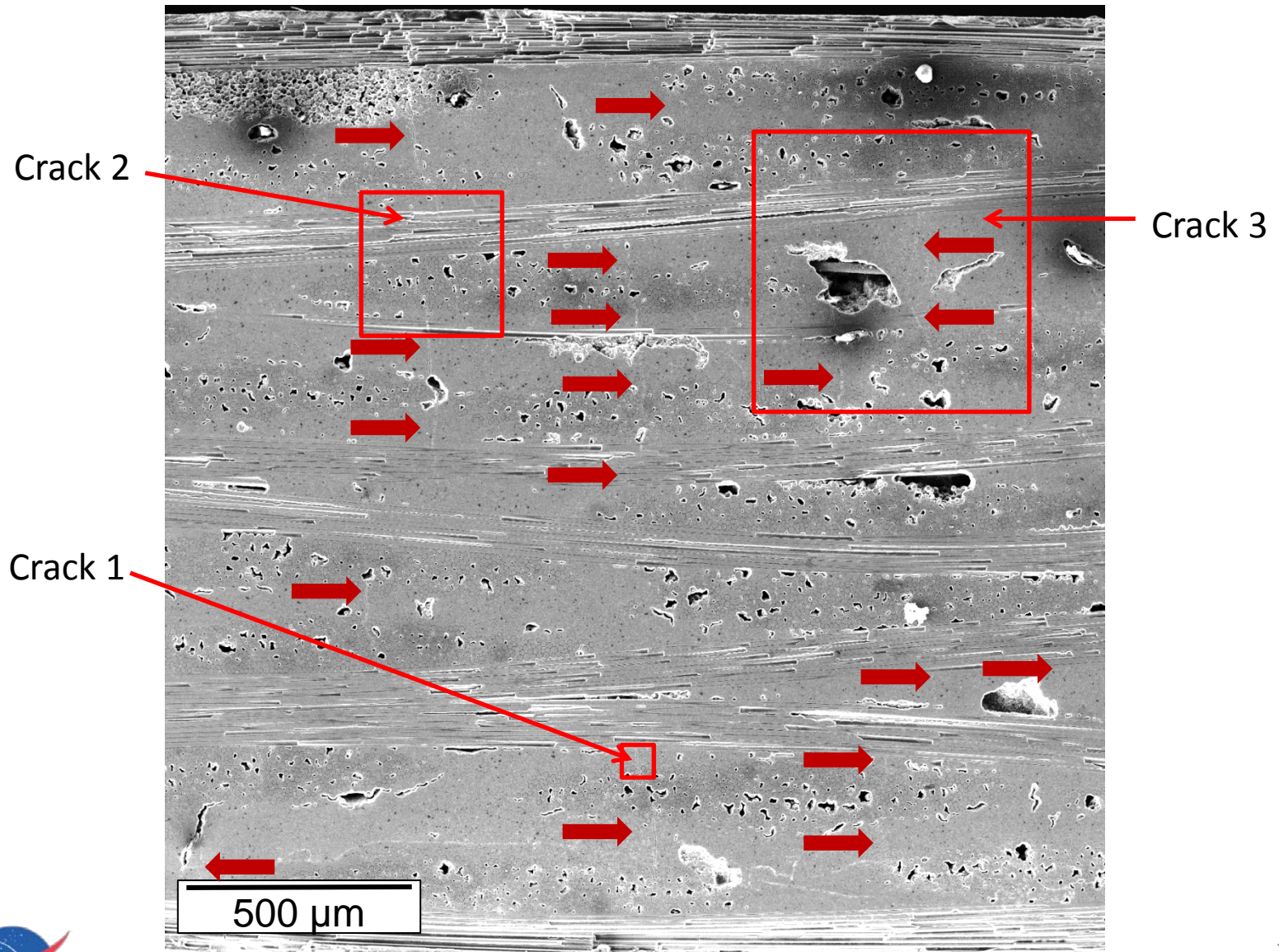
Prior to first
matrix cracking



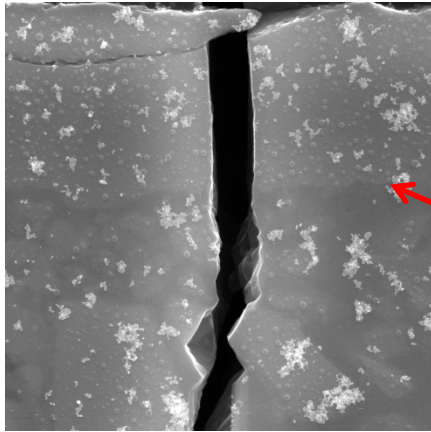
After first
matrix cracking



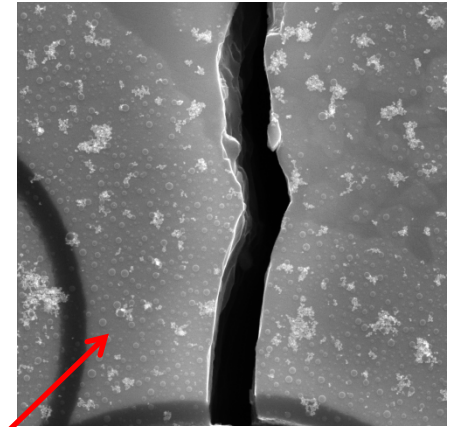
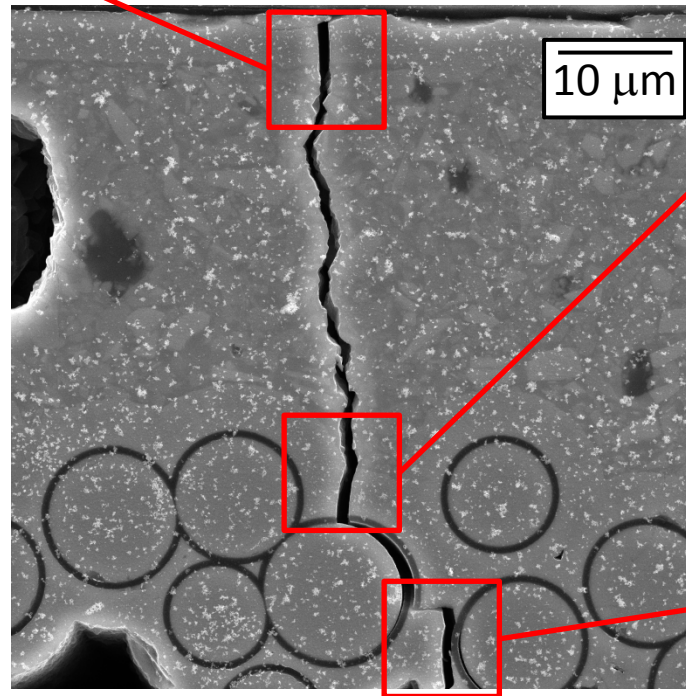
Cracks observed across the cross-section



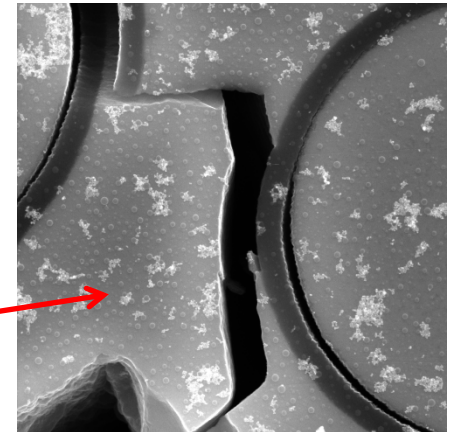
Crack 1



Location 2



Location 1



Location 3

- All high mag FOVs are 10 μm
- High mag FOVs shown at ~30ksi

$\sigma \sim 10$ ksi

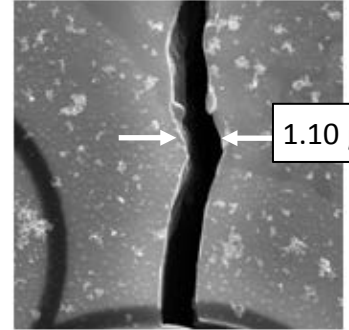
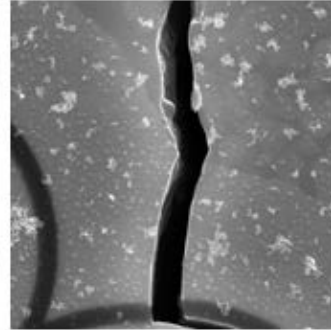
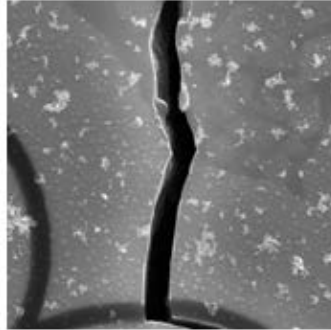
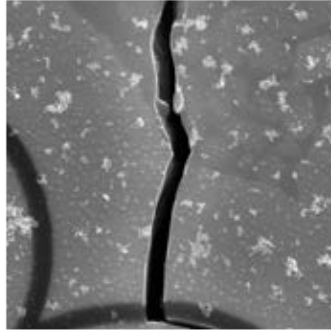
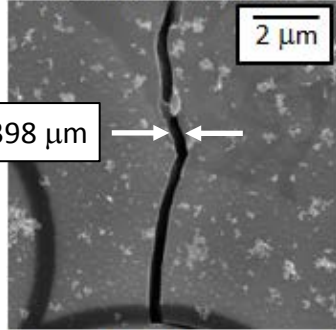
$\sigma \sim 15$ ksi

$\sigma \sim 20$ ksi

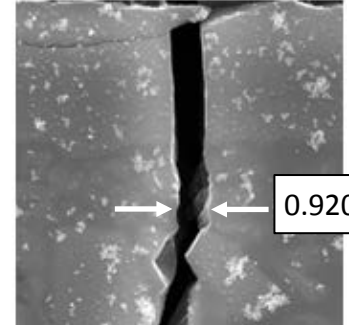
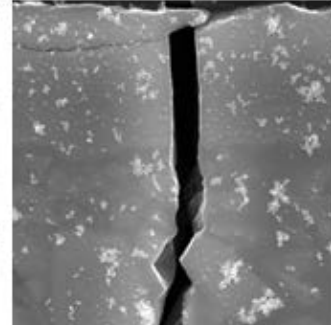
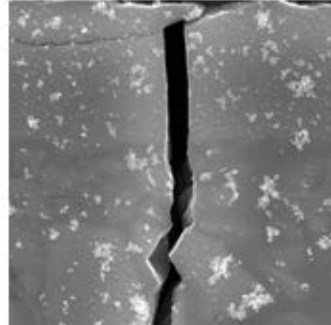
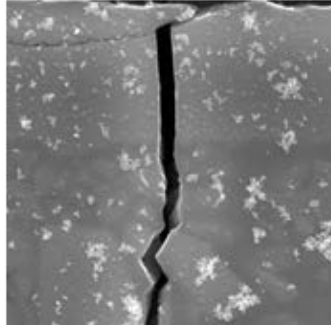
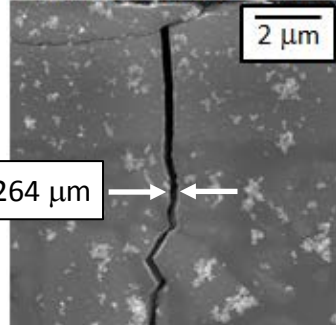
$\sigma \sim 25$ ksi

$\sigma \sim 30$ ksi

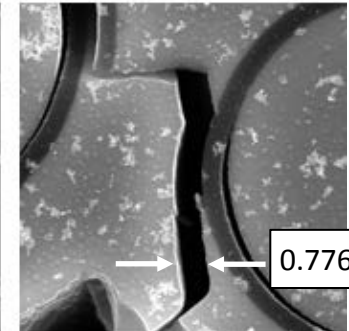
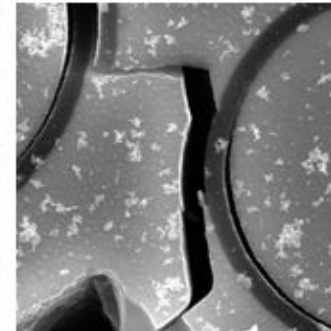
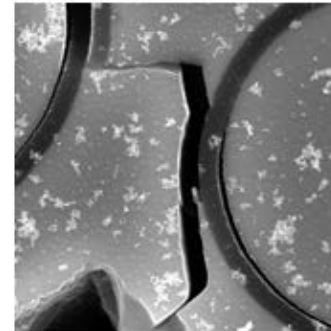
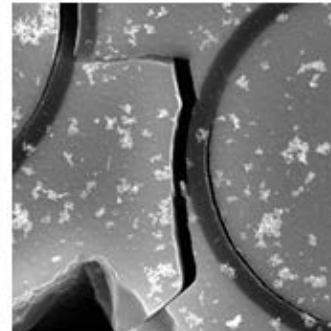
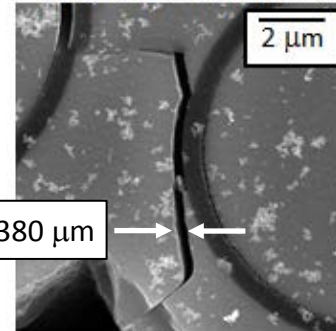
Crack 1 Location 1



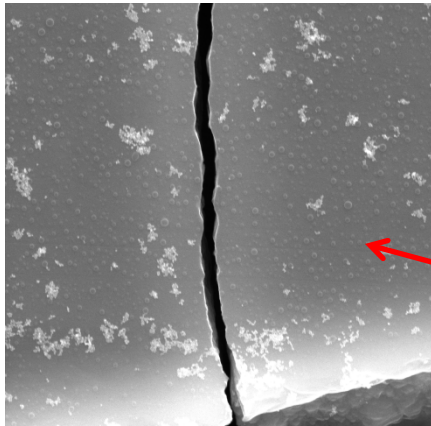
Crack 1 Location 2



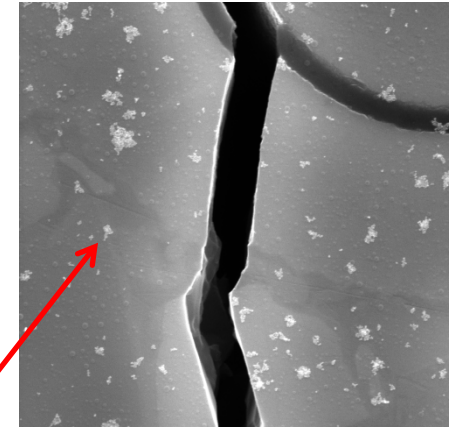
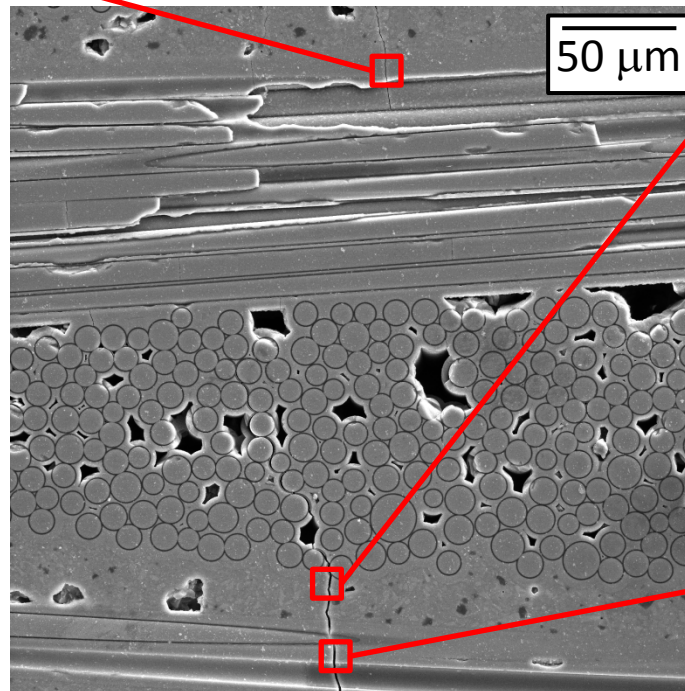
Crack 1 Location 3



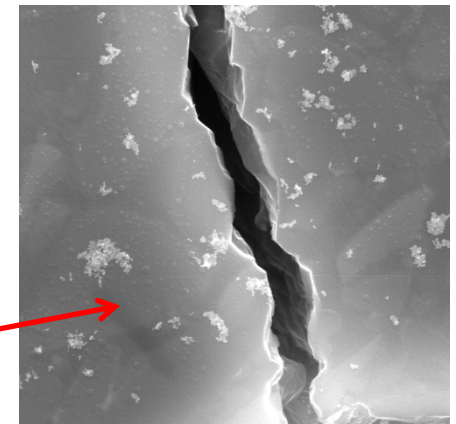
Crack 2



Location 3



Location 2



Location 1

- All high mag FOVs are 10 μm
- High mag FOVs shown at ~30ksi

$\sigma \sim 10$ ksi

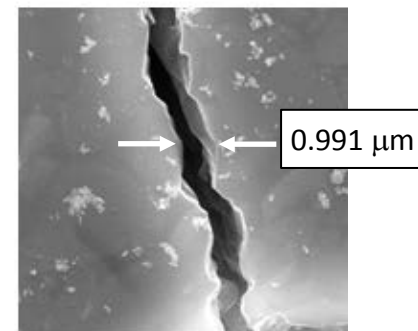
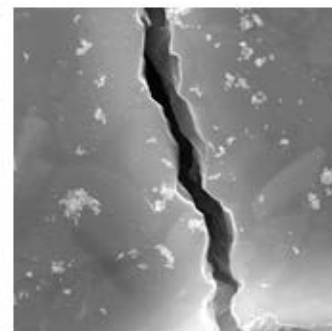
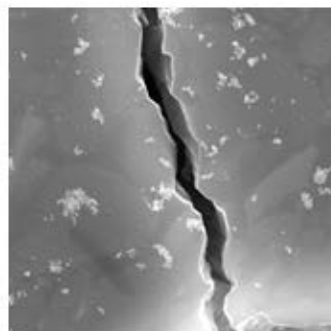
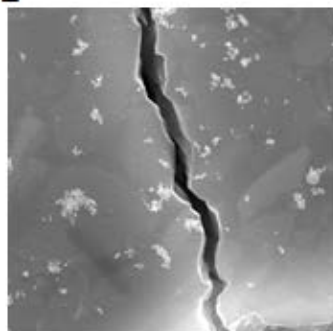
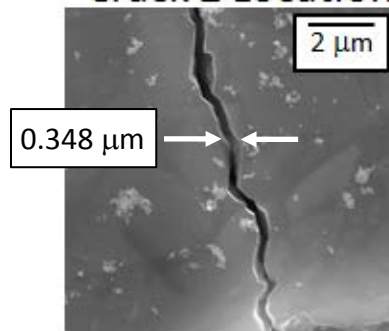
$\sigma \sim 15$ ksi

$\sigma \sim 20$ ksi

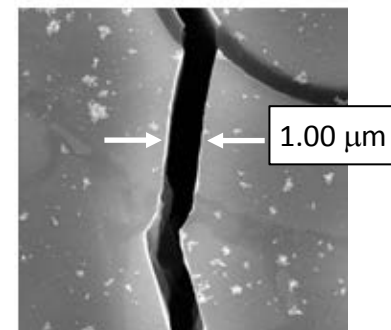
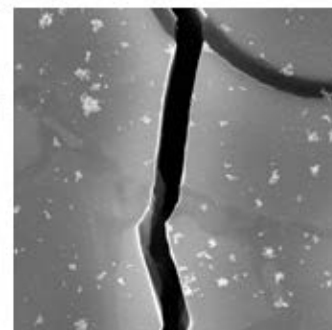
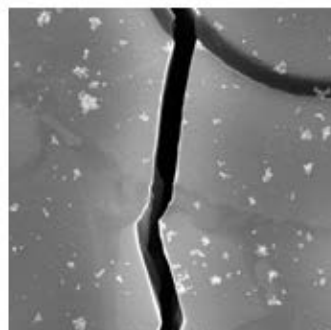
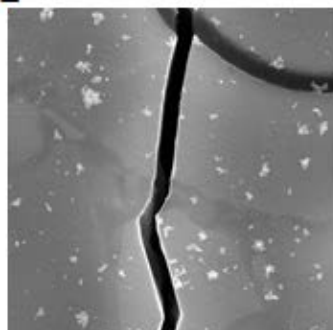
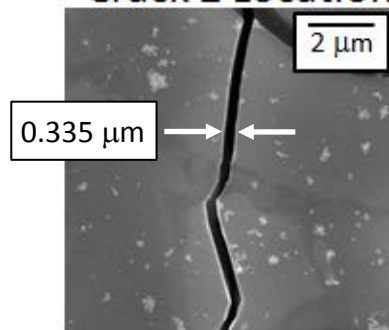
$\sigma \sim 25$ ksi

$\sigma \sim 30$ ksi

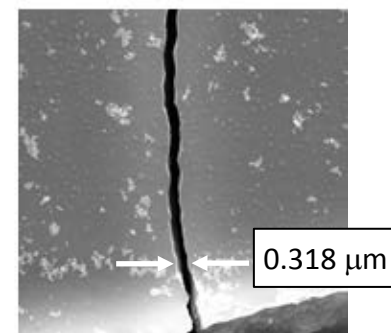
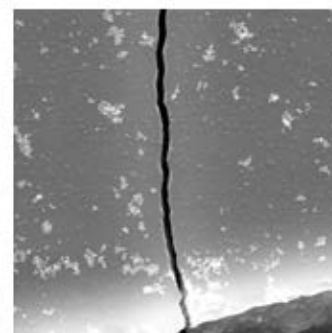
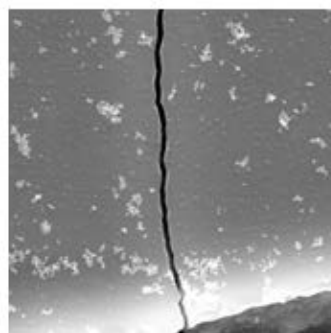
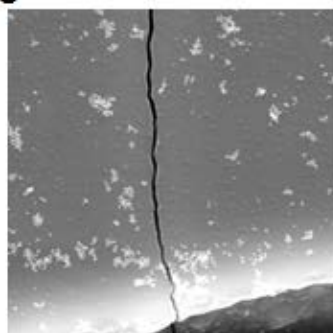
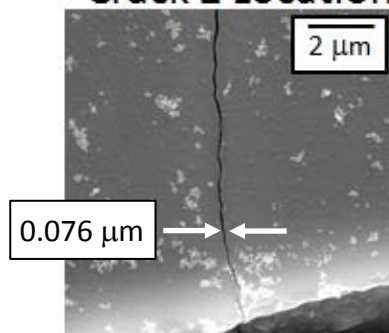
Crack 2 Location 1



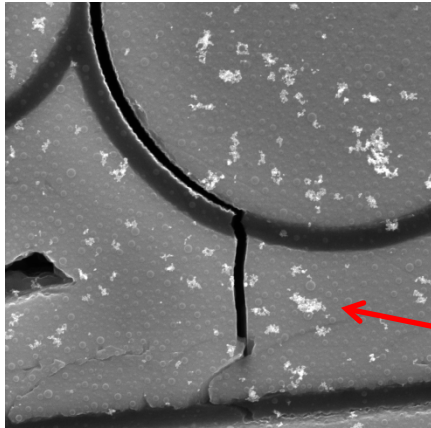
Crack 2 Location 2



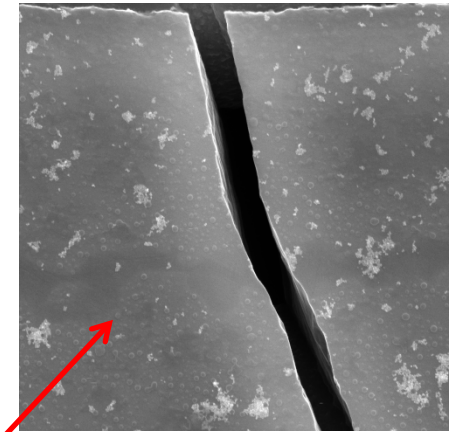
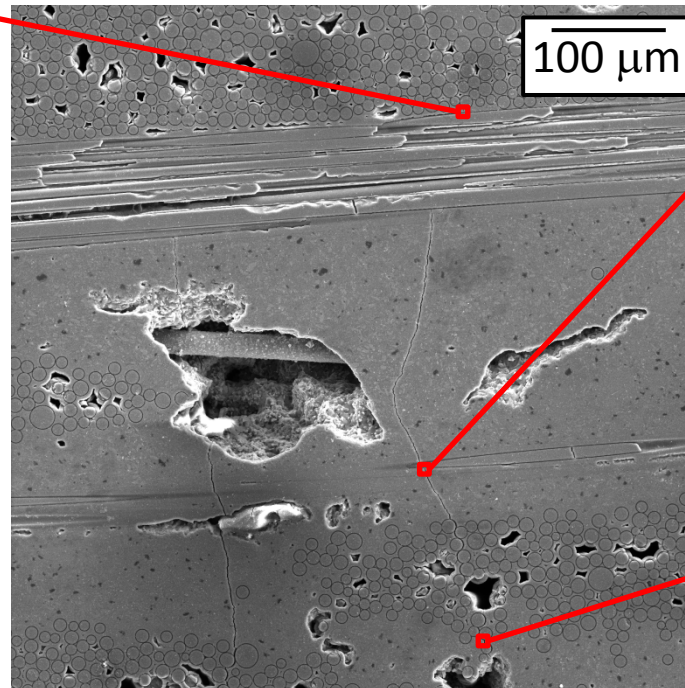
Crack 2 Location 3



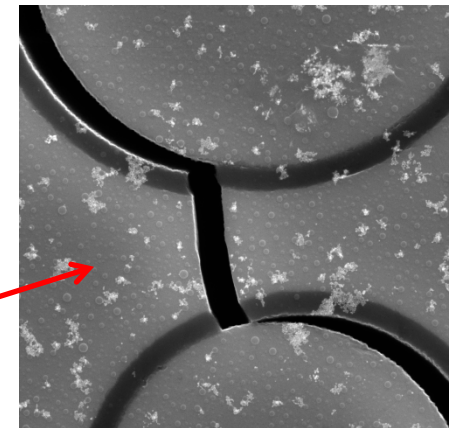
Crack 3



Location 3



Location 2



Location 1

- All high mag FOVs are 10 μm
- High mag FOVs shown at ~30ksi

$\sigma \sim 10$ ksi

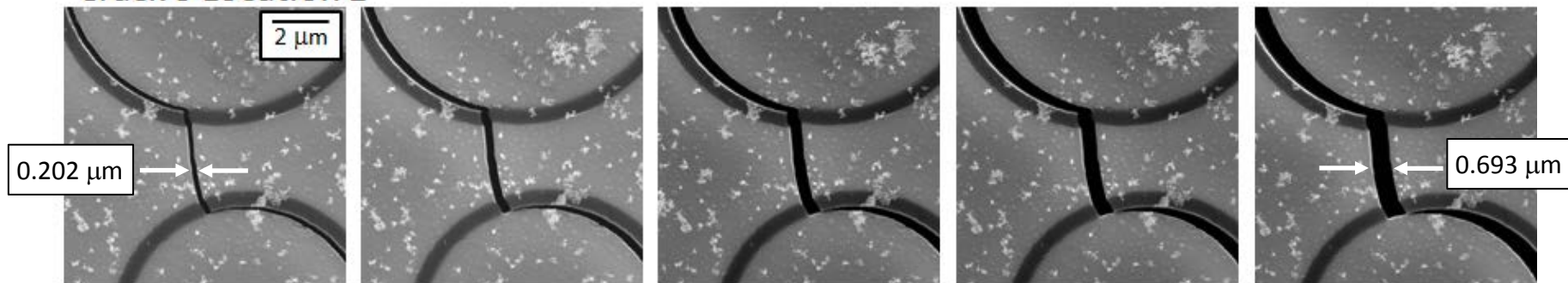
$\sigma \sim 15$ ksi

$\sigma \sim 20$ ksi

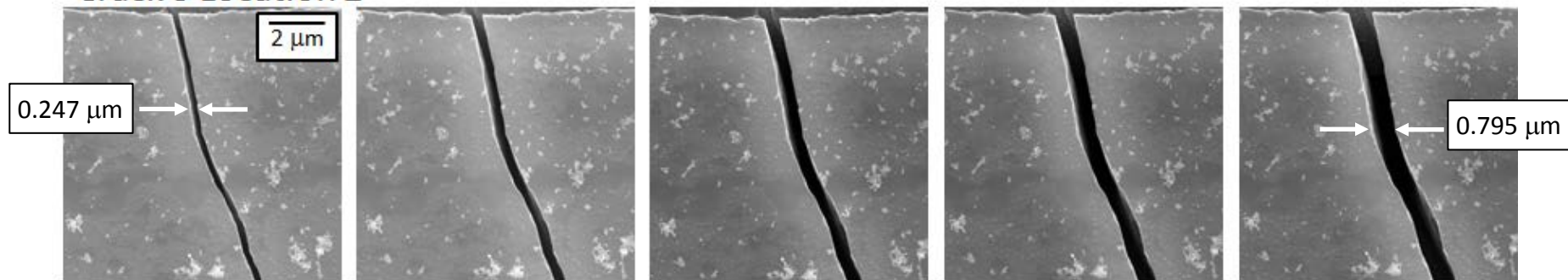
$\sigma \sim 25$ ksi

$\sigma \sim 30$ ksi

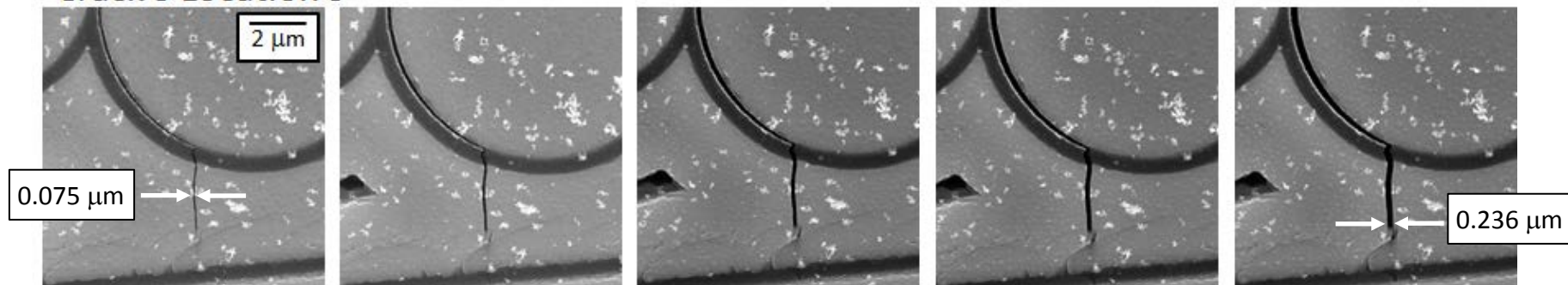
Crack 3 Location 1



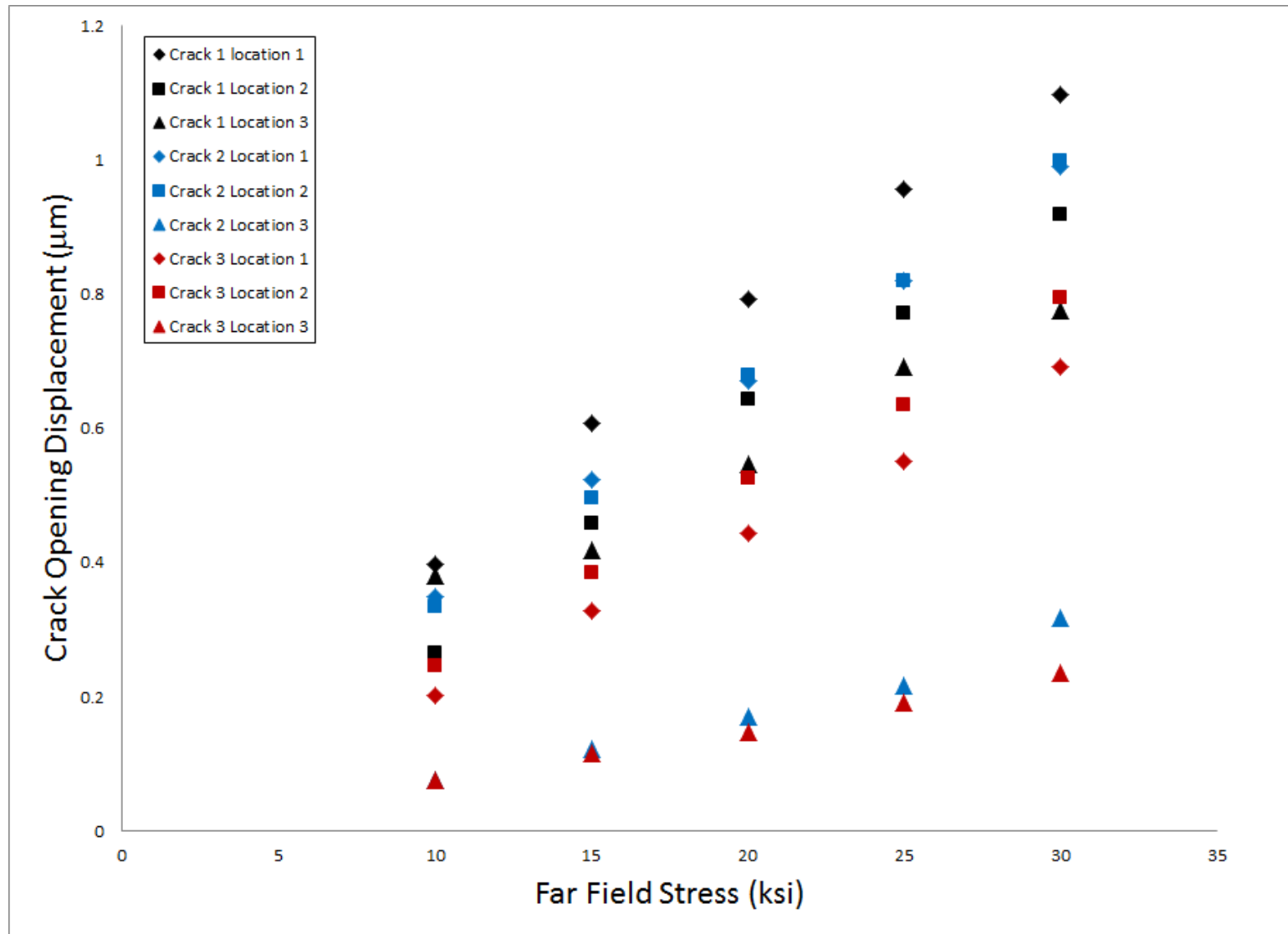
Crack 3 Location 2



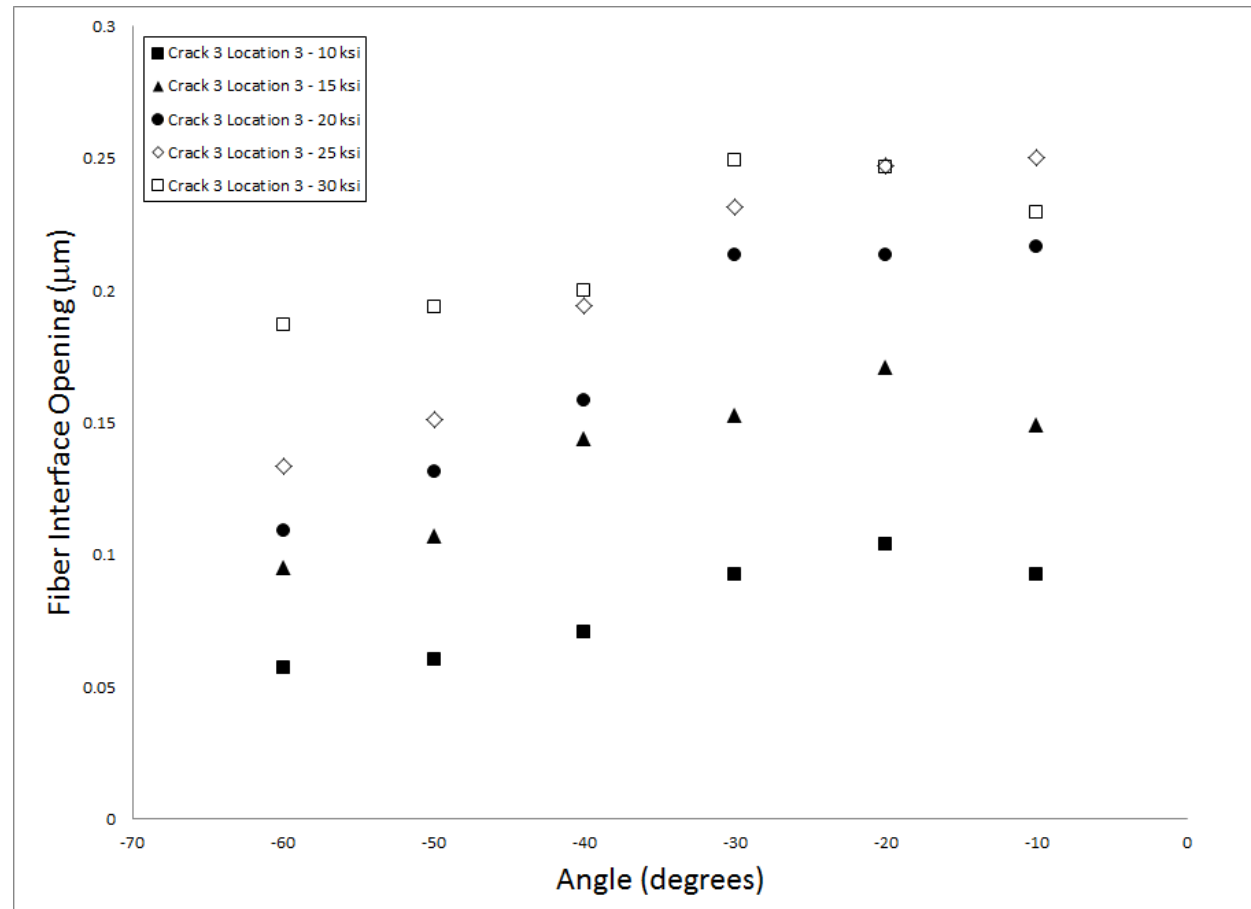
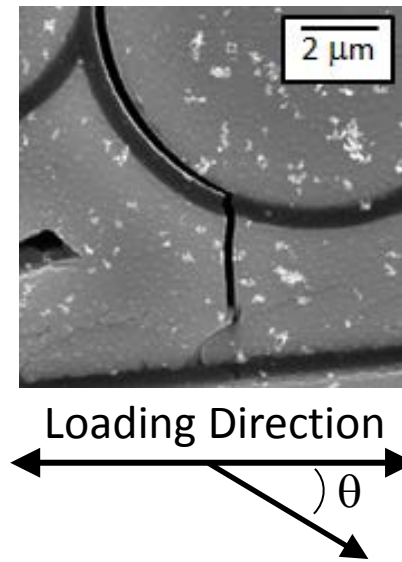
Crack 3 Location 3



Matrix Crack Opening Exhibits Variability



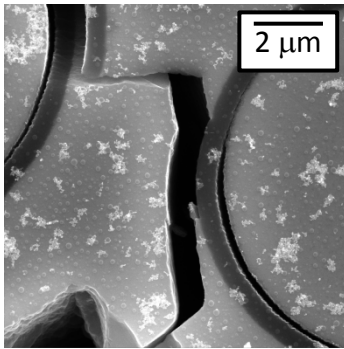
Interface Opening Exhibits Variability



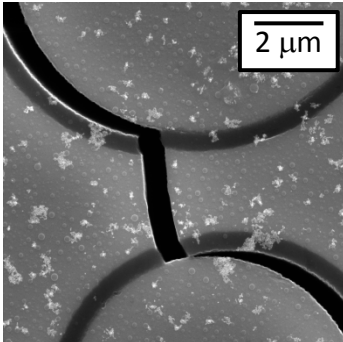
- Expect opening max along direction of stress max
- Stress component along opening direction = $\sigma \cos(\theta)$

Cracking Along Interfaces

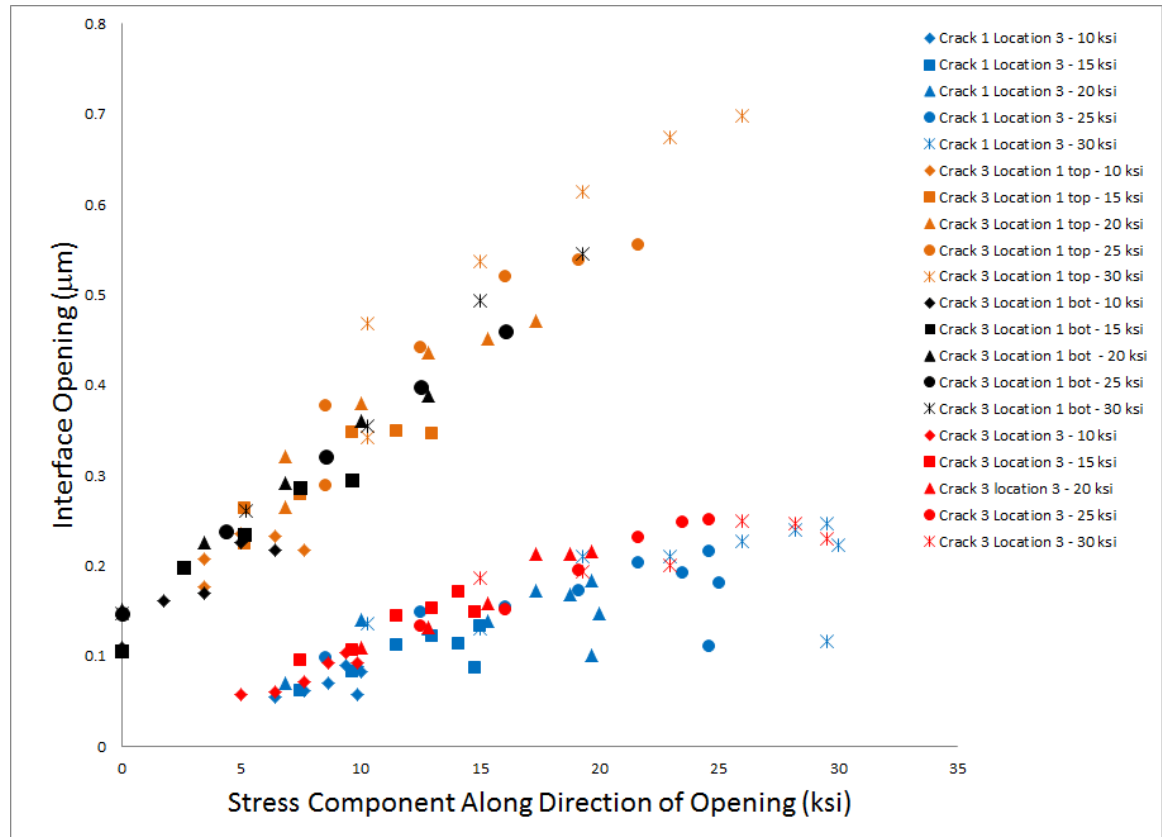
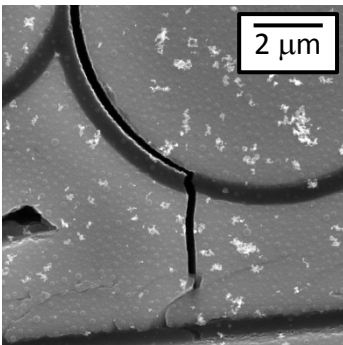
Crack 1 Location 3



Crack 3 Location 1

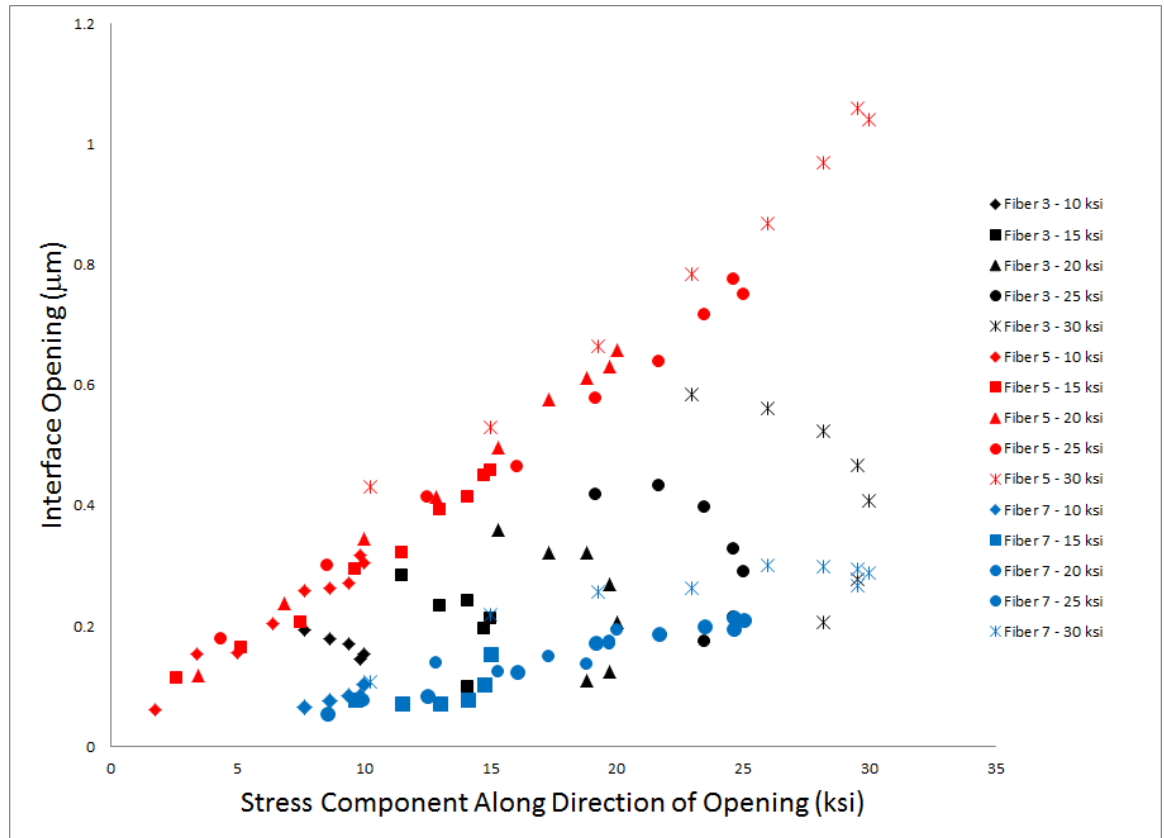
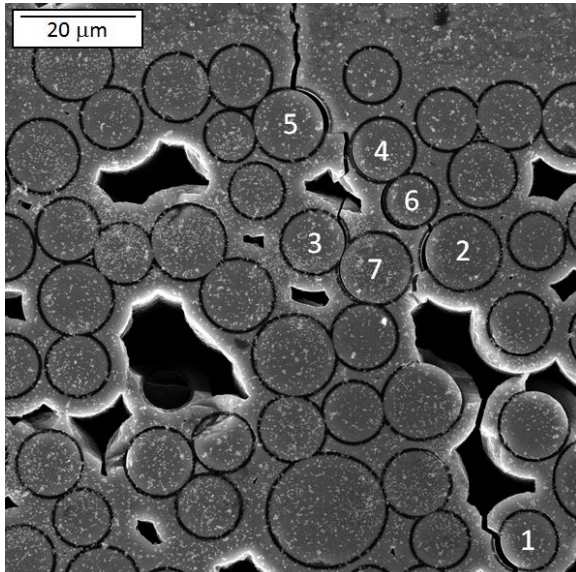


Crack 3 Location 3



- Some openings follow max global stress, some do not
- Cannot see the entire opening in the FOV

Multiple Fibers Along Crack 1



- Again some openings follow max global stress, some do not
- Local stress state is unknown

Future Work

In-situ Testing and Analysis

- Couple macroscale DIC with SEM-DIC to examine the multiscale nature of damage evolution and the influence of microstructure on crack growth
 - Couple high speed imaging with macroscale DIC to examine and quantify the distances over which matrix cracks influence neighboring cracks
- SEM-DIC at ultrafine length scales (FOVs < 5 μm) to probe mechanical response in matrix constituents – available constituent properties are mostly approximations
- Examine environmental effects on subcritical crack growth
 - investigate the effects of fatigue, humidity, combustion gases on crack growth in both coatings and matrix
 - SEM/ESEM (microscale) or an environmental chamber (macroscale)

Modeling

- Statistical modeling of the influence/impact of microstructural features on damage evolution (for data collected in all of the above studies)
 - Quantify and correlate measurements of microstructural features with damage observations
 - Use results to develop models describing the influence of microstructure on damage evolution.



Summary and Conclusions

- A slurry cast MI SiC/SiC sample was loaded to a global stress of 30 ksi in a small tensile stage within an SEM
- SEM-DIC and traditional analysis was used to quantified damage
- Damage at fiber/matrix interfaces at global stresses as low as 5 ksi
- After initial matrix cracking, strain relaxation was observed adjacent to matrix cracks
- Crack opening displacements varied from 0.2 to 1.5 μm at a global stress of 30 ksi
- Interface openings exhibited angular variability where maximum opening was not always along the global loading axis - opening may follow a local maximum

